

Nicotine Extraction from Water with Kerosene

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Distribution coefficients were determined for the water-nicotine-Deobase system at temperatures ranging from 5° to 98° C. and concentrations ranging from 1 to 794 grams of nicotine per liter of water. Factors that control the extraction of nicotine from water by kerosene were determined in a 1.875-inch diameter column containing 39 inches of packing material. Temperature was the most important variable. Calculations were made of extraction capacity coefficient and height of transfer unit in order to correlate data with published data for other systems.

THE demand for nicotine exceeds the supply because it can be produced economically only from tobacco trade wastes, such as stems, damaged leaves and sweepings, and the amount of these that can economically be collected and shipped has reached a maximum. The United States Department of Agriculture is studying *Nicotiana rustica* because of its high nicotine content. As part of the nicotine program, studies were conducted at the Eastern Regional Research Laboratory on the first stage of the liquid-liquid extraction process by which nicotine is extracted from water by kerosene. This paper reports the distribution coefficients at various temperatures and concentrations and the data on continuous kerosene-water extraction. On the basis of this information a pilot-plant unit is being constructed.

The principal methods for recovery of nicotine from tobacco are steam distillation (2), water extraction (1), direct solvent extraction of the tobacco (4), and ion exchange (6). The nicotine is recovered from the vapors of steam distillation either by direct absorption in sulfuric acid (7) or by liquid-liquid extraction after condensation. The latter method yields a water solution of about 0.5% nicotine. At this concentration nicotine cannot be separated from water by fractional distillation at atmospheric pressure because nicotine forms an azeotrope with water (12). The azeotrope contains 2.5% nicotine and boils at 99.988° C. Thus, it would require a large number of plates operating at a high reflux ratio to concentrate the nicotine from 0.5 to 2.5% by fractional distillation at atmospheric pressure. The vapor-liquid relation at pressures above and below atmospheric is not known. Nicotine is extracted from the water phase by a two-stage, liquid-liquid extraction process: in the first stage nicotine is extracted from the water by kerosene; in the second stage the nicotine in the kerosene is extracted from it by sulfuric acid, producing 40% nicotine sulfate, which is the concentration of the commercial product.

DISTRIBUTION COEFFICIENT OF NICOTINE BETWEEN KEROSENE AND WATER

Norton (8) studied the distribution coefficient of nicotine between water and kerosene at 25° C. and concentrations of 0.01 to more than 500 grams of nicotine per liter of solution. The work reported here was undertaken to determine the effect of temperature on the distribution ratio.

EXPERIMENTAL. The nicotine was purified by the vacuum distillation of commercial 99% nicotine. The kerosene was

Deobase (a commercial, refined, and deodorized kerosene) having a density of 0.7797 (d_{24}^{24}) and a boiling range of 160° to 250° C.

Nicotine solutions were prepared that ranged from 1.01 to 793.8 grams of nicotine per liter of solution. The temperature range studied was 5° to 98° C. Fifty milliliters of the nicotine solution were measured into a 250-ml., glass-stoppered Erlenmeyer flask and an equal volume of Deobase was added. The flask was stoppered and the stoppers were sealed with rubber collars. The flask was then placed in a constant temperature bath, which was maintained at the desired temperature $\pm 0.2^\circ$. Although other investigators have shown that equilibrium is established in a short time, the flasks were allowed to remain in the bath for at least 24 hours and shaken at intervals. A sample of the water layer was then pipetted off for analysis. The nicotine was determined by precipitation with silicotungstic acid. The concentration of nicotine in the Deobase layer was calculated by difference. Tests indicated that the solubility of Deobase in the water phase was negligible.

In order to make the calculations, it was necessary to investigate the density of nicotine solutions throughout the concentration ranges involved. Table I shows the densities of the original nicotine solutions, determined by means of a pycnometer.

Maximum density occurred at a nicotine concentration of 65%. Skálweit (11) and Sata (9) reported 66 and 70%, respectively. No volume expansion or contraction in volume was noted when nicotine was added to Deobase in increments; and the density of the Deobase-nicotine solutions increased linearly with concentration.

TABLE I. DENSITIES OF WATER-NICOTINE SOLUTIONS

Nicotine Concentration			Nicotine Concentration		
Grams/liter	%	d_{24}^{24}	Grams/liter	%	d_{24}^{24}
1.01	0.101	0.9978	198.3	19.61	1.0109
2.45	0.245	0.9978	250.5	24.71	1.0139
4.99	0.499	0.9981	300.2	29.50	1.0176
7.45	0.745	0.9979	398.3	38.91	1.0237
9.87	0.987	0.9981	496.9	48.21	1.0289
20.0	2.00	0.9986	594.0	57.47	1.0335
30.0	3.00	0.9996	692.8	66.92	1.0353
40.1	4.01	1.0002	793.8	76.84	1.0331
50.2	5.02	1.0009	853	82.8	1.0297
75.2	7.50	1.0025	904	88.8	1.0245
100.0	9.96	1.0042	951	93.6	1.0167
148.4	14.73	1.0074	1007	100.0	1.0071

EFFECT OF CONCENTRATION. Table II shows the data for the distribution coefficient. The concentration of nicotine in the water layer, C , and in the Deobase layer, C' , is expressed in grams per liter of solution. In Figure 1 the distribution ratio, C/C' , is plotted against the logarithm of the concentration of nicotine in the water phase.

The distribution ratio was relatively constant up to a concentration in the water phase of about 1%, or 10 grams per liter. The ratio then increased with the concentration, reached a maximum between 45 and 65% nicotine, and decreased again at the highest concentrations.

Figure 2 shows the effect of temperature on the distribution ratio for various concentrations of nicotine in the original solution. The ratio varied inversely with temperature, approaching a value of approximately 0.2 between 80° and 85° C. for initial concentrations of nicotine between 0.1 and 40%.

RESULTS. At 0.5 to 0.6% nicotine in water, which is the usual concentration in industry, and with equal volumes of solvent

TABLE II. DISTRIBUTION COEFFICIENT OF NICOTINE BETWEEN WATER AND DEOBASE
(C = concentration of nicotine in water layer, grams per liter; C' = concentration of nicotine in Deobase layer)

Concn. of Nicotine in Original Solution, Grams/Liter	5° C.			20° C.			35° C.			50° C.			65° C.			80° C.			98° C.		
	C	C'	C/C'	C	C'	C/C'	C	C'	C/C'	C	C'	C/C'	C	C'	C/C'	C	C'	C/C'	C	C'	C/C'
1.01	0.78	0.23	3.4	0.62	0.39	1.6	0.45	0.56	0.80	0.34	0.67	0.51	0.21	0.80	0.26	0.20	0.80	0.25	0.20	0.81	0.25
2.45	1.49	0.96	1.6	1.05	1.40	0.75	0.73	1.72	0.42	0.55	1.85	0.30	0.60	1.80	0.33	0.45	2.00	0.23
4.99	3.82	1.17	3.3	2.92	2.07	1.4	2.04	2.95	0.69	1.46	3.52	0.41	1.15	3.79	0.30	1.20	3.79	0.30	0.85	4.12	0.21
7.45	3.04	4.40	0.69	2.12	5.31	0.40	1.59	5.86	0.27	1.80	5.57	0.32	1.15	6.27	0.18
9.87	5.70	4.17	1.4	4.05	5.81	0.70	2.87	6.97	0.41	2.33	7.54	0.31	2.50	7.35	0.34	1.53	8.28	0.18
20.0	15.5	4.55	3.4	11.8	8.22	1.4	8.33	11.6	0.72	5.73	14.2	0.41	4.04	15.7	0.27	4.00	15.8	0.25	2.86	16.9	0.17
30.0	23.4	6.70	3.5	17.9	12.2	1.5	12.5	17.4	0.72	8.60	21.1	0.41	7.06	22.6	0.31	5.90	23.6	0.25	3.69	25.7	0.14
40.1	31.8	8.49	3.7	24.9	15.3	1.6	17.3	22.6	0.76	11.7	27.9	0.42	8.81	30.8	0.29	7.70	31.6	0.24	5.67	33.4	0.17
50.2	40.5	9.98	4.1	31.5	18.9	1.7	22.4	27.6	0.81	14.8	34.7	0.43	9.80	39.2	0.25	9.10	39.7	0.23	6.97	41.7	0.17
75.2	62.7	13.1	4.8	50.5	25.3	2.0	35.6	39.4	0.90	22.7	50.9	0.45	15.4	57.2	0.27	13.2	59.1	0.22	10.1	61.7	0.16
100.0	71.3	29.8	2.4	50.2	49.7	1.0	30.7	66.6	0.46	22.0	73.8	0.30	12.9	81.0	0.16
148.4	132	18.4	7.2	112	39.1	2.9	52.8	91.4	0.58	28.7	109	0.26	18.0	117	0.15
198.3	163	40.0	4.1	125	76.6	1.6	73.9	118	0.63	44.8	138	0.32	25.2	151	152	0.15
250.5	232	23.1	10.1	213	44.8	4.8	174	83.8	2.1	107	138	0.78	57.4	169	0.34	38.0	181	0.21	27.0	186	0.15
300.2	226	86.0	2.6	155	145	1.1	64.1	201	0.32	44.3	211	0.21	30.2	217	0.14
398.3	378	30.5	12.4	358	57.2	6.3	327	93.3	3.5	248	164	1.5	108	244	0.44	53.9	273	0.20	35.0	273	0.13
496.9	478	31.7	15.0	461	58.3	7.9	436	92.6	4.7	396	138	2.9	175	278	0.63	64.3	315	0.20	40.7	321	0.13
594.0	573	44.0	13.0	550	84.2	6.5	540	99.6	5.4	514	135	3.8	410	231	1.8	72.6	359	0.20
692.8	669	63.4	10.5	652	99.4	6.6	609	168	3.6	87.4	398	0.22
793.8	761	112	6.8	739	163	4.5	706	219	3.2	658	274	2.4	92.6	435	0.21	53.6	438	0.12

and solution, approximately 48% of the nicotine was removed in one extraction stage at 25° C., whereas at 85°, about 83% was removed. In the solvent extraction of nicotine with kerosene, it is obviously advantageous to operate the extraction columns at high temperature.

EXTRACTION IN A PACKED TOWER

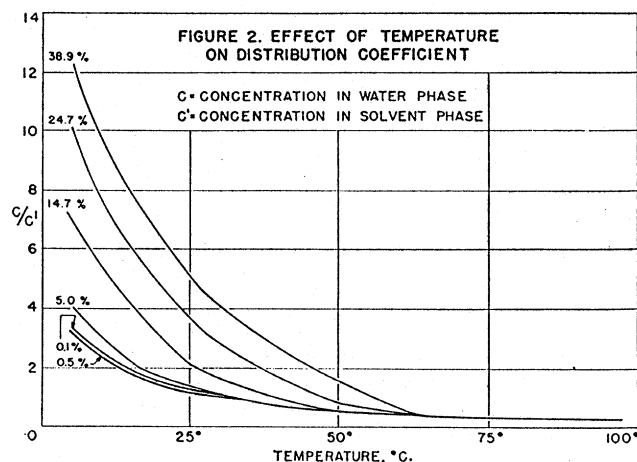
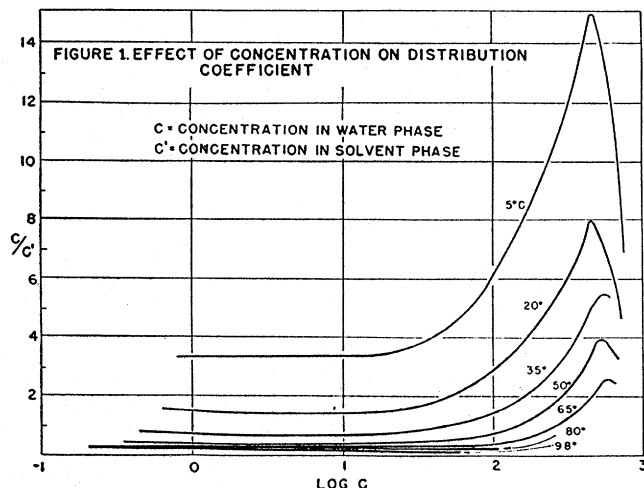
EQUIPMENT. Figure 3 shows a diagram of the extraction equipment. The overhead reservoir, *A*, contained a solution of nicotine in tap water, ranging in concentration from 0.10 to 0.50% by weight. The nicotine solution flowed by gravity from the overhead reservoir through rotameter *B* and heating coil *C* into the top of packed column *D* at *E*, down through the column, leaving at *F*, then through inverted seal *G*, and was discharged at *H*. Kerosene with specific gravity of 0.82 and flash point of 70° C. was stored in the overhead reservoir, *J*. The distribution coefficient of the commercial grade of kerosene was 0.8 at 25° C. The kerosene flowed by gravity through rotameter *K* and heating coil *L*, through the distributor *M*, up through the column leaving at *N*, and was discharged at *O*. The heating coils were copper tubing immersed in a bath of hot water. The piping was made of a polyvinylidene chloride plastic. Tests showed there was no loss of nicotine in contact with this material. By using this semitransparent tubing, air pockets could be detected; on several occasions these interfered with steady flow conditions.

Although rotameters were used to regulate flows, all data on flows reported here are volumetric measurements of the two streams leaving the column.

The packed column was made of 1.875-inch inside diameter glass tubing 48 inches long. The upper 6 inches and lower 3 inches of the column had no packing. The packed section was 39 inches long, with 0.0624 cubic feet of free space. The kerosene distributor, *M*, was made from a 0.25-inch I.P.S. cap with 7 holes of 0.0625-inch diameter in the top. Figure 4 shows a sketch of this distributor.

In most of the runs water was the continuous phase and kerosene was the dispersed phase; the interface was held in the upper 6 inches of the column (above the packing) by regulating value *P* (Figure 3) and the height of seal *G*. In runs in which water was the dispersed phase and kerosene the continuous one, the water was atomized by a distributor similar to *M*, and the interface level was controlled in the lower 3 inches of the column by regulating valve *P* and seal *G*. The column was insulated, but because it was not jacketed true isothermal conditions were not obtained. A thermometer was installed in each of the two inlet streams to the column. The temperature of the streams was the same and was maintained constant within $\pm 2^\circ$ C.

Column packing was: (A) 0.25-inch ceramic Berl saddles; (B) 0.375-inch ceramic Raschig rings (0.375-inch outside diameter \times 0.25-inch inside diameter \times 0.375-inch long); (C) 0.5-inch Berl saddles; and (D) 0.5-inch Raschig rings (0.5-inch outside diameter \times 0.3125-inch inside diameter \times 0.5-inch long).



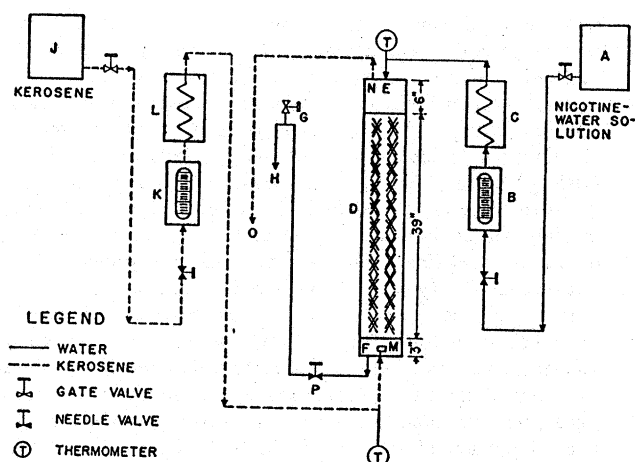


Figure 3. Extraction Equipment

The effects of loose and tight packing of the Berl saddles were also investigated. As in industrial practice, to decrease breakage the loose packing was made by dropping the saddles into the column full of water. The tight packing was made by dropping the saddles into an empty column and then tapping.

OPERATION. The column was filled with water preferentially to wet the packing. The two streams (nicotine-water solution and nicotine-free kerosene) were then passed through the column. After the flow rates, temperatures, and interface level were adjusted, the column was operated at steady flow conditions for sufficient time to replenish the continuous phase at least three times. Volumetric measurements of the two effluent streams were made and samples taken. The maximum flow rates reported for each series of runs are the rates just below flooding. With kerosene as the dispersed phase, the column floods at the bottom, both phases passing out the bottom instead of water only. With water as the dispersed phase, the column floods at the top. The flooding rates are approximate only (probably $\pm 5\%$). Because the column becomes unstable as the flow rates approach flood conditions, it would be unduly time-consuming to attain precise flood conditions.

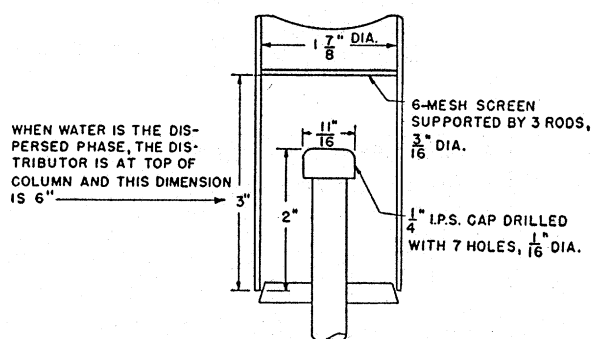


Figure 4. Distributor for Dispersed Phase

The water solutions were analyzed by titrating with 0.1 *N* hydrochloric acid; methyl red was used as an indicator. Methyl red was chosen because its color-change point, pH 4.8 to 5.4, is close to the pH of nicotine monosulfate. The latter is given by Norton (8) as 5.5; potentiometric titrations at this laboratory (13) gave approximately 5.2. To determine nicotine in the kerosene solutions, water in excess was added, and the same titration was made, with constant shaking. The color change in this case was not sharp; as the reaction occurs in water solution, it cannot proceed any faster than the extraction permits. Moreover, the amount of nicotine extracted by the kerosene was

usually less than the amount extracted from the water, as shown by titrations of the water. The low concentrations of the solutions in many cases made the titrations difficult; some were as dilute as 0.05% nicotine, or a mole fraction of 0.000056. Several samples of water solutions were checked by Willits *et al.* by the silicotungstic acid method of the Association of Official Agricultural chemists. Agreement was within 0.01% of nicotine. The same was true of seven samples of kerosene solutions not actually produced in this work but similar; these were the only ones subjected to a check.

The results, tables, and graphs given in this paper are based solely on the water analyses. Because the accuracy of the kerosene analyses is somewhat doubtful, the nicotine content of the kerosene was calculated from the amount of nicotine removed from the water. The kerosene analyses are useful as a rough check. The equilibrium concentration of the water phase at the top of the column was obtained by multiplying the calculated nicotine content of the kerosene at that point by the appropriate distribution coefficient taken from Figure 1. In the graphs, the concentration of nicotine in the water inlet is 0.50%, kerosene is the dispersed phase, and the volume ratio of kerosene to water is 1.

RESULTS. Figure 5 shows the percentage of nicotine removed from the water at 21° C. with three types of packing. Listed in the order of increasing efficiency, they were: 0.25-inch Berl saddles packed tight, 0.375-inch Raschig rings, and 0.25-inch Berl saddles packed loosely. These results show that the industrial method of dropping the saddles into the column full of water gives higher extraction.

Figure 5 also shows the percentage of nicotine removed from water at 63° C. The 0.25-inch Berl saddles packed loosely gave slightly higher extraction than the 0.375-inch Raschig rings, and with both these packings the extraction decreased with increasing flow. The 0.5-inch saddles loosely packed gave 5% higher extraction than 0.5-inch rings, and with either, no decrease of extraction occurred with increased flow. The flooding rates with both 0.5-inch packings were much higher than with 0.25- or 0.375-inch packing.

Figure 6 shows the effect of temperature on percentage of nicotine removed. The upper curve shows the nicotine extraction in the column with 0.25-inch Berl saddles packed loosely and water flow rate of 21 cubic feet per hour per square foot. The lower curve is a replot of the distribution coefficient of the system taken from Table II. The curves are approximately parallel, showing the value of distribution coefficients in predicting good extraction. Temperature was the most important factor in obtaining high extraction efficiency. Although extraction at 21° was 28%, this was increased to 92% at 84° C., other conditions being equal.

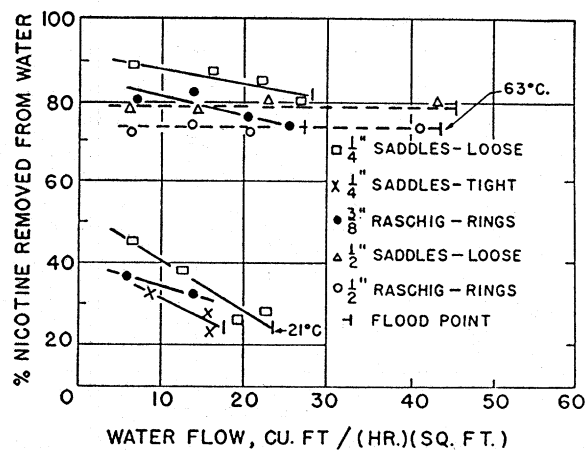


Figure 5. Effect of Water Flow on Removal of Nicotine

TABLE III. PERCENTAGE OF NICOTINE REMOVED WITH 0.25-INCH BERL SADDLES IN EXTRACTION COLUMN

Water Feed		Nicotine, %	<i>E/W</i> ^a	Temp., ° C.	Phase Dispersed	Nicotine Removed, %
Rate, Cu. Ft./ (Hr.) (Sq. Ft.)						
A. Effect of Concentration of Nicotine in Feed						
11.8	0.50	1.00	21	Kerosene	38	
13.2	0.36	1.11			36	
15.3	0.24	0.95			37	
14.6	0.15	1.00			33	
14.6	0.10	1.05			35	
B. Effect of Kerosene to Water Ratios, <i>E/W</i>						
11.8	0.50	1.00	21	Kerosene	38	
6.6		1.79			56	
6.6		3.06			72	
C. Effect of Phase Dispersed						
6.3	0.50	1.56	66	Kerosene	88	
16.0		1.09			86	
22.3		1.00			84	
26.5 ^b		1.05			80	
66	0.50	1.37	66	Water	90	
16.0		1.04			82	
22.3		1.00			76	
34.2 ^b		1.27			64	

^a *E/W* = volume ratio of kerosene to water.
^b Flow rate was just below flooding rate.

Table III, A, shows that the percentage of nicotine extracted was practically constant over the range of 0.10 to 0.50% nicotine in the water inlet to the column.

As was expected, greater extraction of nicotine was obtained with higher kerosene-to-water ratios by volume, *E/W* (Table III, B). Most published work on liquid-liquid extraction includes tests on high ratios. In this work the ratio (*E/W*) was maintained at approximately 1.0, except in this series of runs. The ratio of 1.0 was dictated by the following considerations: If the concentration of nicotine in water were 0.50% and *E/W* were 4, for example, the kerosene would contain about 0.15% nicotine by weight. In commercial practice, such dilute solutions would throw the load on the second extraction stage, where the nicotine is extracted from kerosene with sulfuric acid.

Table III, C, shows that at low flow rates high extraction is obtained, whichever phase is dispersed, but if water is the dispersed phase the extraction falls off more rapidly as the flow rate is increased, and the flooding rate is higher.

Nicotine extraction could be duplicated within 2% as long as the column was clean but was erratic when the column was fouled. An emulsion formed at the interface between the water and kerosene. If this emulsion was allowed to accumulate on the packing, the pattern of the kerosene drops ascending through the water was changed. With a clean column, the drops of kerosene ranged from 0.125 to 0.25 inch in diameter

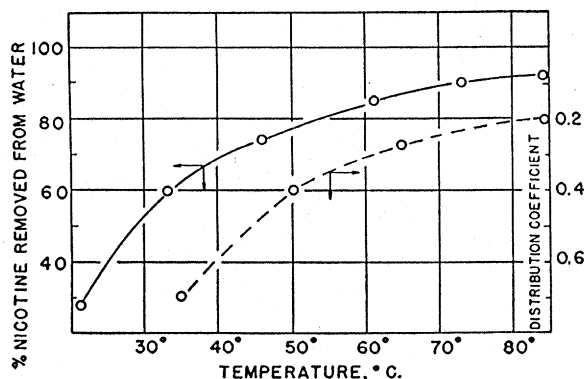


Figure 6. Effect of Temperature on Removal of Nicotine and on Distribution Coefficient

and ascended comparatively rapidly; with a fouled column however, the kerosene ascended more slowly in a rolling motion and in irregularly shaped drops as large as 0.5 inch.

Some difficulty was also experienced because of settling of the 0.25-inch Berl saddles that packed loosely in the column. After standing for several weeks the voids had decreased, indicating settling of the packing. The percentage of nicotine extracted decreased with the more densely packed saddles. The ring type of packing will be used in the pilot plant unit, as these showed no evidence of settling. The ring type gave 5% lower efficiency, but reproducibility of results is more important.

In order that the data reported here may be correlated with published data for other systems, the extraction capacity coefficients and the heights of transfer unit have been calculated for a few of the runs. These are calculated separately for each phase, but the calculations are based on the gross volume of the packed portion of the column in both cases. The extraction capacity coefficient in the water phase is designated $K_w a$, and that in the kerosene phase K_a . The height of the transfer unit in the water phase is designated HTU_{ow} , and that in the kerosene phase, HTU_{oa} . The calculations are based on the method of Elgin and Browning (3) and Johnson and Bliss (5). The formulas used are:

$$K_w a = \frac{N/\theta}{(V)(\log \text{mean } \Delta C)}$$

and

$$HTU_{ow} = \frac{L_w}{K_w a}$$

$K_w a$ = over-all extraction coefficient based on the water side, lb. moles/(hr.) (cu. ft.) (unit ΔC)

N/θ = moles of nicotine transferred per unit time, lb. moles/hr.

V = effective volume of the column, considered to be the empty volume of the packed section, cu. ft.

Log mean ΔC = log mean of C_1 and C_2

C_1 and C_2 = concentration difference at the two ends of the column, $C-C^*$, lb. moles/cu. ft.

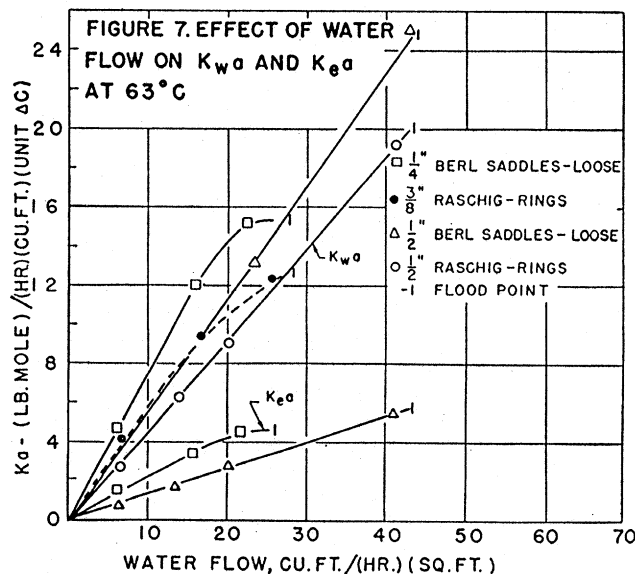
C = actual nicotine concentration in water, lb. moles/cu. ft.

C^* = concentration of nicotine in water which would be in equilibrium with observed concentration in kerosene, lb. moles/cu. ft.

HTU_{ow} = over-all height of a transfer unit, based on the water phase, ft.

L_w = flow rate of water phase, cu. ft./(hr.) (sq. ft.)

Figure 7 shows that the coefficient $K_w a$ varied with water flow rates (continuous phase) for four types of packing. The 0.25-inch Berl saddles gave the highest coefficient, which was



pected, because this size is within the minimum 8 to 1 ratio of column diameter to packing size generally used in distillation and absorption. The 0.5-inch saddle type gave a higher coefficient than the ring type of the same size; this was in agreement with Sherwood *et al.* (10). However, the difference in the coefficients was small and, on the industrial scale, the choice between ring and saddle type of packing could well be made on the relative costs per cubic foot of the two packings.

Figure 7 also shows that the coefficient K_a was much smaller than the K_{wa} . The water-nicotine-kerosene system had appreciable resistance to extraction in both the water and kerosene phases, and neither resistance could be ignored. The kerosene phase offered the major resistance. The kerosenes were then modified by the addition of oil-soluble surface-active agents to reduce the surface tension of the kerosene and thus reduce the resistance to extraction of the kerosene phase, thereby increasing the over-all extraction. The following surface-active agents were added in a concentration of 0.1%: calcium stearate, Nopalcol 1-L (a nonionic mono-lauric derivative), and Nopalcol 1-O (a nonionic mono-oleic derivative). A dispersing agent was also added to the water (0.1% of Daxad No. 11, a sodium salt of a short-chain alkyl naphthalene sulfonic acid). However, none of these four agents increased extraction of nicotine from the water phase.

Figure 8 shows the effect of extraction temperature on K_{wa} and K_a when 0.25-inch Berl saddles and a water flow of 21 cubic feet per hour per square foot were used. No published data on other systems could be found showing the effect of temperature on the individual coefficients; hence a comparison cannot be made. It is interesting that one coefficient can increase rapidly with temperature while the other coefficient remains constant. The pronounced effect of temperature can be seen clearly in this graph; a sevenfold increase in the coefficient K_{wa} was obtained by raising the extraction temperature from 21° to 84° C. From the point of view of nicotine chemistry, it adds support to the

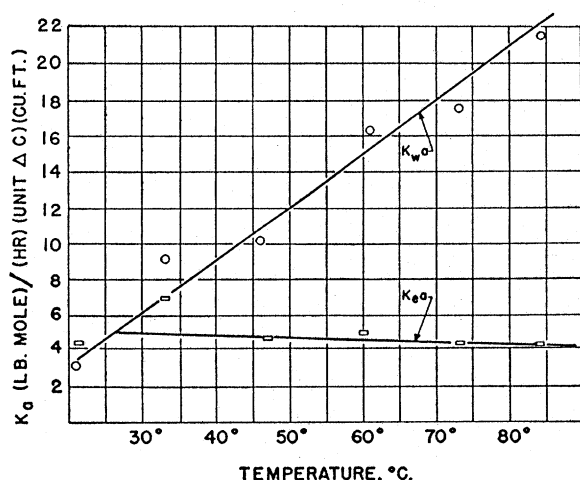


Figure 8. Effect of Temperature on K_{wa} and K_a

TABLE IV. HTU and K_a WITH KEROSENE DISPERSED AND 0.50% NICOTINE IN WATER

HTU = over-all height of a transfer unit; subscript *ow* refers to water phase; subscript *oe* refers to kerosene phase
 K_a = extraction capacity coefficient; subscript *w* refers to water phase; subscript *e* refers to kerosene phase

Temp., ° C.	Flow Rate, Cu. Ft./ (Hr.) (Sq. Ft.)		Ratio of Kerosene to Water	K_a , Lb. Mole/(Hr.) (Cu. Ft.) (ΔC)		HTU, Ft.	
	Water	Kerosene		K_{wa}	K_{ea}	HTU_{ow}	HTU_{oe}
	EFFECT OF TEMPERATURE						
21	22.3	15.4	0.69	3.2	4.2	6.9	3.7
33	22.3	19.6	0.88	9.1	7.0	2.4	2.8
46	19.6	20.2	1.03	10.2	4.7	1.9	4.2
61	22.3	22.3	1.00	16.4	5.1	1.4	4.4
73	21.0	22.3	1.06	17.4	4.3	1.2	5.2
84	23.8	22.3	0.95	21.5	4.3	1.1	5.2
EFFECT OF PACKING							
0.25-Inch Berl Saddles Packed Loosely							
63	6.3	9.8	1.56	4.7	1.3	1.6	7.5
63	16.0	17.5	1.09	12.0	3.6	1.3	4.9
63	22.3	22.3	1.00	15.1	4.5	1.5	5.0
63	26.5	27.9	1.05	15.4	4.6	1.7	6.0
0.375-Inch Raschig Rings							
63	7.0	8.4	1.20	4.0	1.2	1.8	7.0
63	14.0	16.8	1.20	8.4	2.5	1.7	6.7
63	20.4	20.9	1.02	10.3	3.1	2.0	6.7
63	25.1	27.9	1.11	12.3	3.6	2.0	7.8
0.5-Inch Berl Saddles							
63	6.6	9.1	1.38	3.4	1.0	1.9	9.1
63	14.7	16.8	1.14	7.8	2.3	1.9	7.3
63	23.0	24.4	1.06	13.3	3.8	1.7	6.4
63	43.2	34.2	0.79	24.9	7.7	1.7	4.4
0.5-Inch Raschig Rings							
63	6.3	9.1	1.44	2.7	0.8	2.3	11.4
63	13.3	15.4	1.16	6.2	1.8	2.1	8.6
63	20.0	23.7	1.18	8.9	2.6	2.3	9.1
63	41.2	44.0	1.07	19.4	5.6	2.1	7.9

theory of a nicotine hydrate, reasoning from the following: over-all extraction increases with temperature; the increased extraction with temperature is not due to the kerosene phase but is confined to the water phase.

Table IV shows the over-all height of the transfer unit, based on both the water and kerosene sides. The height equivalent to a theoretical stage (HETS) was also estimated by graphic methods. In most of the runs, however, the number of theoretical stages was less than one, and because with less than one stage the HETS method of evaluating the data is only approximate, these data have not been reported.

A pilot plant unit is being designed based on the data obtained in this work. A 4-inch column packed to a height of 12 feet with 0.5-inch Berl saddles should remove about 98.5% of nicotine, based on either coefficient—the K_{wa} or K_a .

SUMMARY

Maximum density of water-nicotine solutions occurred at 65% nicotine, whereas the density of kerosene-nicotine solutions increased linearly with concentration. The distribution coefficient (ratio of nicotine in the water layer to nicotine in the kerosene layer) was constant at any one temperature up to a concentration of 1% nicotine in the original solution. The coefficient decreased with increasing temperature at all concentrations; in concentrations below 1%, it decreased from 3.4 at 5° C. to 0.2 at 85° C. At constant temperatures, the coefficient increased with concentrations, reached a maximum between 50 and 70% nicotine, and decreased again at higher concentrations.

By increasing the temperature from 21° to 84° C., extraction of nicotine from water in a packed column was increased from 28 to 92%. Extraction was increased about 8% by packing the saddles loosely instead of tightly. The saddle type of packing gave about 5% higher nicotine extraction than the same size of ring packing. Flood rates with either the 0.5-inch ring or saddle packing were about 45 cubic feet per hour per square foot, each, of water and kerosene, whereas with 0.25-inch saddles or 0.375-inch rings the flood rate was about 28 cubic feet per hour

per square foot. At low flow rates, the 0.25-inch saddles resulted in about 10% higher nicotine extraction than 0.5-inch saddles, but at higher flow rates, extraction was practically the same with either size. Extraction was essentially constant over the range of 0.1 to 0.5% nicotine in water as the feed stock. Extraction was increased about twofold by tripling the ratio of kerosene to water. When high flow rates were used, extraction was increased about 16% by dispersing the kerosene rather than the water; at low flow rates, however, extraction was the same with either phase dispersed.

The extraction capacity coefficient at 63° C., based either on the water or kerosene phase, increased with increasing flow rates, but the coefficient based on the water phase was 3.5 times that based on the kerosene side—that is, kerosene offered the major resistance in this system. The coefficient based on the water phase increased uniformly with temperature, increasing sevenfold over the temperature range 21° to 84° C., whereas the coefficient based on the kerosene side remained constant over this temperature range.

The height of the transfer unit varied with temperature, flow rate, and other operating conditions but covered the range of 1.1 to 6.9 feet when based on the water side, and from 2.8 to 11.4 feet when based on the kerosene side.

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LITERATURE CITED

- (1) Aitken, R. G., Can. Patent 401,143 (Dec. 2, 1941).
- (2) *Chem. & Met. Eng.*, **42**, 373 (1935).
- (3) Elgin, J. C., and Browning, F. M., *Trans. Am. Inst. Chem. Engrs.*, **31**, 639 (1935).
- (4) Fritzsche, R., *Syracuse Chemist*, **35**, 3 (1942).
- (5) Johnson, H. F., and Bliss, H., *Trans. Am. Inst. Chem. Eng.*, **42**, 331 (1946).
- (6) Kingsbury, A. W., Mindler, A. B., and Gilwood, M. E., *Chem. Eng. Progress*, **44**, 497 (1948).
- (7) McConnell, H. K., U. S. Patent 1,927,180 (June 28, 1924).
- (8) Norton, L. B., *IND. ENG. CHEM.*, **32**, 241 (1940).
- (9) Sata, Naoyasu, *Bull. Chem. Soc. Japan*, **2**, 139 (1927).
- (10) Sherwood, T. K., Evans, J. E., Longcor, J. V. A., *IND. ENG. CHEM.*, **31**, 1144 (1939).
- (11) Skilweit, J. J., *Ber.*, **14**, 1809 (1881).
- (12) Smith, C. R., *IND. ENG. CHEM.*, **34**, 251 (1942).
- (13) Willits, C. O., *et al.*, Eastern Regional Research Laboratory, private communication.